

## **Guidance - Mercury Monitoring to Evaluate Amalgam Rule Implementation**

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The Department issued letters to many dischargers in late 2007 and early 2008 requiring specialized mercury effluent monitoring to enable the Department to evaluate the effectiveness of the implementation of the 'Amalgam Rule'. The following attachments are provided to assist in implementing this project:

**Attachment 1 - Frequently Asked Questions.** Questions concerning these Frequently Asked Questions may be directed to Muhammad Shaikh in the Bureau of Point Source Permitting – Region 1 at (609) 633-3869.

**Attachment 2 – Sample mercury monitoring letter.** The letter contains the specific requirements of this monitoring project.

**Attachment 3 - A list of current certified laboratories.** Any questions concerning certified laboratories may be directed to the Office of Quality Assurance at (609) 633-3840. Please note that this list is current as of early 2008 and permittees may check for any updates at the following website:

[http://datamine2.state.nj.us/DEP\\_OPRA/OpraMain/categories?category=Certified+Laboratories](http://datamine2.state.nj.us/DEP_OPRA/OpraMain/categories?category=Certified+Laboratories)

**Attachment 4 - Clean Hands/Dirty Hands Technique.** This document provides additional technical sample collection guidance for the required Clean Hands/Dirty Hands Technique. Any questions concerning this attachment may also be directed to the Office of Quality Assurance at (609) 633-3840.

Note: This document is specific to wastewater effluent testing and sample collection at the request of the Department. The user is instructed to read this **entire** document before planning a sample collection event. Each sampling team is encouraged to practice the Clean Hand/Dirty Hand technique described in this attachment prior to sample collection.

## **Attachment 1**

### **Frequently Asked Questions**

#### **1. What are some sources of sample contamination and how to avoid it?**

There are numerous routes by which samples may become contaminated. Potential sources of trace metal contamination during sampling include metallic or metal-containing sampling equipment, containers, labware (e.g. talc gloves that contain high levels of zinc), reagents, and deionized water; improperly cleaned and stored equipment; and atmospheric inputs such as dirt and dust from automobile exhaust, cigarette smoke, nearby roads, bridges, wires, and poles. Even human contact can be a source of trace metals contamination. For example, it has been demonstrated that dental work (e.g. mercury amalgam fillings) in the mouths of laboratory personnel can contaminate samples that are directly exposed to exhalation. Samplers must wear a mask during sampling and/or sample equipment preparation. Masks with activated carbon filters are recommended to maximize the protection of the sample, but dust masks are also approved for use.

Clean, non-talc polyethylene, latex, vinyl or PVC gloves are required during the handling of sample bottles and sampling equipment. In addition to the wrist length gloves required, shoulder length gloves are also needed for sample collection by direct submersion of the bottle for the Clean Hands (CH) member of the sampling team. The Dirty Hand (DH) member of the sampling team may wear the wrist length gloves only.

Fluoropolymer (commonly known as Teflon<sup>®</sup>) or borosilicate glass containers with fluoropolymer or fluoropolymer lined caps (and fluoropolymer tubing if a peristaltic pump is going to be used for sample collection) must be utilized for the collection of field blanks and samples.

All sample containers (and sampling equipment if provided by the certified laboratory) must be certified as clean before being shipped from the certified laboratory to the sampling site in accordance with the requirements of EPA Method 1631E (Section 6.1.2.1, sample bottles) and EPA Method 1669 (Section 6.15.2 for tubing if a peristaltic pump will be used for sample collection).

Documentation regarding the certification of mercury-free sample containers and sampling equipment must be retained on file at the laboratory or facility for review by the Department upon request. If sampling equipment is cleaned at the sampling site prior to sample collection, then the sampling team performing the cleaning must also retain documentation regarding the reagents (lot number of acid used) and materials used, either at the laboratory or facility, for review by the Department upon request including the results of the equipment blanks required in Item 7 below.

Sample collection bottles and sampling equipment materials should not be stored in excess of the amount of materials required for the project. Prolonged periods of storage may potentially expose the bottles and sampling materials to contamination.

The samples are to be collected during periods of representative flows (no collection at extreme peak flows or during storm or high wind events).

## **2. What type of sample is to be collected?**

The USEPA currently recommends that samples collected for low level mercury testing be grab samples as compositors may be more subject to sources of contamination. Therefore for this project, all effluent samples shall be **grab** samples.

If desired by the permittee, mercury monitoring can be performed on the influent in addition to the required effluent sampling for informational purposes. Influent samples are not required for this project. **If the influent is sampled, all blanks and effluent samples must be collected first to avoid potential carry over contamination. Clean Hands/Dirty Hands sampling techniques should be employed as discussed in Attachment 4. Influent samples must be clearly identified as such so that the laboratory may take any necessary precautions to eliminate cross sample contamination or instrument contamination.**

**Note: Unless the permittee has historical data verifying that the influent sample results are Non-detect (ND) at 0.2 µg/L, the permittee should consider first analyzing the influent sample by the less sensitive method, EPA Method 245.1. Influent samples with potential mercury concentrations greater than 0.1 µg/L could result in sample cross contamination and require sample dilutions by the laboratory if analyzed by EPA Method 1631E. Influent samples should be discussed with your contract laboratory.**

## **3. How many samples are to be collected?**

In the first round of sampling three effluent samples shall be taken, one sample at a time collected at least 30 days apart. This sampling **does not** have to be initiated within 30 days of the letter and the sampling can start at any date as long as the entire first round of samples is collected by June 30, 2008. As an example if samples were taken on April 29<sup>th</sup>, May 29<sup>th</sup> and June 30<sup>th</sup> (June 29<sup>th</sup> is a Sunday) this sampling schedule would meet the requirements of this project. It is best however to begin sample collection at the beginning of a month. If the last sample is to be collected on June 30<sup>th</sup> for example, and a storm event or a period of high winds occur on that day, the sample can not be taken and you would not be able to meet the requirements of the project. The Department also recommends that these sampling events be handled as a separate project and not performed in conjunction with other routine effluent monitoring events as protection of the samples from mercury contamination is paramount.

The second round of sampling shall be between the period of February 1, 2010 and July 30, 2010. The requirements for sample collection above also apply to the second round of sampling required.

**4. What analytical method is required?**

Samples shall be analyzed for Total Mercury using only EPA Method 1631E by a laboratory certified by NJDEP to perform that method. See **Attachment 3** for a listing of these laboratories. A link to the method is provided below:

<http://www.epa.gov/waterscience/methods/method/mercury/1631.pdf>

**5. Is EPA Method 1669 (Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, EPA 821-R-96-011, July 1996) required for sample collection?**

Yes. Section 6.8.2.2.8, Trace Element Sampling, of the NJDEP's Field Sampling Procedures Manual requires the use of the USEPA recommended Method 1669 clean sample techniques to ensure the elimination of potential contamination from the actual sample collection process.

The ease of contaminating water samples with the metal(s) of interest and interfering substances cannot be overemphasized. EPA Method 1669 includes sampling techniques that should maximize the ability of the sampling team to collect samples reliably and eliminate sample contamination. The dirty hand (DH) – clean hand (CH) procedure is required and is addressed in **Attachment 3**.

A link to the method is provided below:

<http://www.epa.gov/waterscience/methods/method/inorganics/1669.pdf>

**6. Are field blanks required?**

Yes. A field blank sample must be collected at the same site at the same time as each effluent sample. The testing laboratory must provide all of the distilled water for use in this sampling effort. The source of laboratory water must be demonstrated to be free from contamination. This distilled water must be used to perform the field blank and to rinse sampling equipment prior to sample collection. The laboratory shall include as part of the labeling for the distilled water container, the date of its preparation by the laboratory doing the sample analysis (testing laboratory). Documentation must be retained on file at the laboratory and must be made available to the Department if requested.

Field blank sample bottles must include the date of collection on the labels provided by the testing laboratory or at a minimum on the sample collection log and must be directly linked to the effluent sample collected.

The field blank sample must be collected in the same manner as the effluent sample. An aliquot of the laboratory provided distilled water must be transferred from the testing laboratory- supplied container of distilled water to an empty sample container (if sample is simply dipped into the water stream or collected by a sampling pole with bottle support) or processed through the tubing of a peristaltic pump system (if used). The field blank sample bottle is filled prior to collecting the effluent sample. The field blank sample must then accompany the effluent sample to the testing laboratory.

The field blank and effluent sample must be collected directly into the sample container provided by the testing laboratory. No dippers or intermediate containers are allowed. Field blanks must be collected before the effluent samples.

## **7. Are Equipment Blanks required?**

If the sampling equipment (**distinguished from the sample bottles which must be supplied by the certified laboratory performing the analytical testing**) is provided to the permittee or contracted sampling party by the certified laboratory performing the analytical testing, then equipment blanks (EPA Method 1669, Section 9.3) are the responsibility of that certified laboratory. Records establishing compliance with the requirements of EPA Methods 1631E or 1669 for sampling equipment must be retained on file by the laboratory and must be made available to the Department upon request.

If the permittee or contracted sampling party is preparing the sampling equipment for the project, equipment blanks are required to be collected and tested for each piece of sampling equipment that will be used. The equipment blanks must show that the sampling equipment is free of mercury contamination **prior to its use** in any sampling event in accordance with EPA Method 1669, Section 9.3.5.

## **8. How are the samples preserved?**

Samples are to be preserved by the testing laboratory. Samples must be preserved or analyzed within 48 hours of sample collection. Once properly preserved (within the initial 48 hours of sample collection), the holding time for the testing can then be extended to 90 days. If not preserved or tested within 48 hours, the sample must be rejected and re-sampled.

## **9. How is data to be reported?**

**Blank correction (subtraction of field or equipment blanks from the sample results) is prohibited at any time.** Sample results and the results of the field blank analysis (and equipment blanks if required) must be reported separately.

Laboratory reports showing the results of all 3 sets of samples and blanks analyzed in the first round shall be submitted to the NJDEP in one package within 30 days of the end of the first round of sampling. The same requirement applies to the second round

of sampling and analysis to be performed in 2010. **Results shall NOT be entered onto monitoring report forms (i.e. DMR, WCR, RTR).**

Data shall be submitted to:

Muhammad Shaikh, Supervising Environmental Engineer  
NJDEP Division of Water Quality  
Bureau of Point Source Permitting-Region 1  
P.O. Box 029  
Trenton, New Jersey 08625-0029

## **Attachment 2**

### **Sample Mercury Letter**

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Subject: Monitoring Requirements for Mercury using Method 1631E for NJPDES Permit No. NJ00XXXXX

Dear Permittee:

As you may be aware, the Department has implemented a multi-faceted approach to controlling and reducing mercury released in the air, soil and waters of our state. In order to reduce mercury levels into our surface and groundwaters, the Division of Water Quality has adopted a new section in the NJPDES regulations at N.J.A.C. 7:14A-21.12, called the Amalgam Rule. This rule establishes best management practices and regulatory requirements for owners of dental facilities that generate amalgam waste through the removal or placement of amalgam fillings and requires dentists to install amalgam separators. The expected result is a reduction in the amounts of mercury amalgam released into the collection system and ultimately into the waters of the state.

In order to determine the effluent base line levels of mercury discharged from the state's sewage treatment plants the Department, pursuant to the authority contained in N.J.A.C. 7:14A-6.2(a)4.i, 6.2(a)14, and N.J.A.C. 7:9A-1.5(e)7, is requiring that you conduct certain effluent testing for mercury, using EPA method 1631E.

By requiring this testing for mercury both before and after implementation of the Amalgam Rule, The Department will be able to better ascertain the impact of this rule and determine if further actions need be undertaken. Method 1631E is an EPA approved method for detecting the presence of mercury in wastewater effluent. The Method Detection Level (MDL) is 0.02 ng/L for this method. However, for the purposes of this testing, the Recommended Quantification Level (RQL) shall be 1.0 ng/L. This testing is described in detail below: Testing Protocol: Three samples must be taken as early as possible, however, at intervals no less than 30 days, from the date of this letter through June 30, 2008.

Three additional samples must be taken, at intervals no less than 30 days apart, during the period of February 1, 2010 through July 30, 2010 (6 samples total). Samples must be taken in accordance with the Department's current Field Sampling Procedures Manual. However, due to a current lack of New Jersey Laboratories certified for Method 1631E, the Department will accept results from out-of-state laboratories, provided they are NJ Certified.

Submission of the lab test result sheets within 30 days apart of the completion of each of the 2 rounds of sampling to Muhammad Shaikh in the Department's Bureau of Point Source Permitting – Region 1 at the address listed above shall be considered compliance with this letter. Please do not enter the results on your monitoring report forms (eg. DMR, WCR, RTR).

For questions pertaining to your NJPDES Discharge to Surface Water Permit, please contact the permit writer listed on your latest permit action or if you believe you have received this letter in error, please contact Muhammad Shaikh as above.

For questions pertaining to information related to the Office of Quality Assurance please contact Joseph Aiello at (609) 633-3840.

Please note that failure to comply with this requirement is a violation of N.J.A.C. 7:14A-6.2(a)4.i and 14 and will result in enforcement action.



## **Attachment 3**

### **Laboratories Certified to Perform Mercury Analysis Using Method 1631E**

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| Lab Number | Lab Name                                | Phone Number |
|------------|---|--------------|
| FL006      | TESTAMERICA PENSACOLA                   | 850-471-6242 |
| ME002      | KATAHDIN ANALYTICAL SERVICES, INC.      | 207-874-2400 |
| NY004      | COLUMBIA ANALYTICAL SERVICES, INC.      | 585-288-5380 |
| OH001      | TESTAMERICA NORTH CANTON                | 330-966-7290 |
| OH006      | SUMMIT ENVIRONMENTAL TECHNOLOGIES, INC. | 330-253-8211 |
| PA011      | LANCASTER LABORATORIES, INC             | 717-656-2300 |
| SC002      | GEL LABORATORIES, LLC                   | 843-556-8171 |
| WA004      | BATTELLE MARINE SCIENCES LABORATORY     | 360-681-4550 |
| WA005      | COLUMBIA ANALYTICAL SERVICES INC        | 360-577-7222 |
| WA007      | FRIEDMAN & BRUYA INC                    | 206-285-8282 |
| WA010      | BROOKS RAND LABS                        | 206-632-6206 |

# Attachment 4

## Grab Mercury Sample Collection Guidance Using Clean Hands/Dirty Hands Technique

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### I. Preliminary Preparation (required days before the actual sampling event)

- Laboratory cleans fluoropolymer or borosilicate sample collection bottles, double bags them in clean, zip-type, non-vented, colorless polyethylene bags and ships them to the client.
- Laboratory or sample collectors clean and double bag sampling equipment.
- Laboratory or sample collectors prepare the fluoropolymer tubing which must be cleaned by donning disposable powder-free gloves, and filling the tubing with a 50% trace element grade nitric acid solution, immersing tubing into a 10% HCL bath for a minimum of seven days. The tubing is then purged with mercury-free air or nitrogen in a metal free area and double bagged in new zip-type, non-vented, colorless polyethylene bags.
- Sampling poles and bottle supports must be cleaned with nitric acid and rinsed with mercury-free water, dried in an area known to be free of mercury contamination sources, double-wrapped with clean, colorless polyethylene, and stored before use.

**Note: If bottles and/or sampling equipment arrive in ripped plastic bags, do not proceed with the sampling. Notify the laboratory, certified cleaning contractor or sampling contractor and make arrangements for another bottle and/or sampling equipment shipment.**

### II. Sampling Packages

- Laboratory or sample collectors prepare sampling packages in designated clean non-metallic, white interior cooler containing:
  - Cleaned and double-bagged sample tubing if a peristaltic pump will be used. Clean and double-bagged pole with bottle support if used for sample collection.
  - Clean, zip-type, non-vented, colorless polyethylene bags, dust masks, non-talc clean gloves.
  - Plastic sheet (clean, colorless polyethylene) for ground cover
  - Sample bottles (provided by the testing laboratory, prepared by documented procedures, see Sections 6.1 and 6.2 of EPA Method 1631E).
- Bag with extra gloves for site preparation activities.
- Peristaltic pump with easy load head if used. Extension cord, if necessary.
- Or, metal free bottle support designed to hold the sample bottles provided by the laboratory performing the testing. Bottle clamps must be made of stainless steel or fluoropolymer plastic. **Neither the pole nor the pump is required if a representative sample can be collected by simply dipping the sample bottle into the effluent flow.**

- Waste bucket.
- Sample storage cooler, clean, non-metallic, white interior.
- Carboy or large fluoropolymer or borosilicate bottle with distilled water determined to be free of contamination by the laboratory for the field blank.

### **III. Site Preparation**

- Upon arrival at the sampling site, the sample collectors don the Tyvek suits (if used), and unloads equipment and prepares for sample collection.
- Samples must not be taken in areas where dirt and dust from automobile exhaust, nearby roads, bridges, wires, and poles will potentially contaminate the sampling process.
- Samplers must not breathe directly over the samples. It has been demonstrated that dental work (e.g. mercury amalgam fillings) in the mouths of laboratory personnel can contaminate samples that are directly exposed to exhalation.
- Samplers must not smoke at the sampling location or in any area where the field blank, sample or sampling equipment is handled.
- Anything covering the sample site, such as a grate or manhole, is removed and set aside.
- CH and DH waits for 10 minutes before beginning sampling process to allow the sampling environment to stabilize.
- CH and DH don the required gloves and activated carbon or dust masks.
- Plastic is laid out for ground cover, with coolers on the ends to secure.
- The peristaltic pump is set up (if used) or the sampling pole is assembled and left covered in the inner protective layer of plastic wrap until use.
- Clean Hands (**CH**) and Dirty Hands (**DH**) sample collectors get into position.

### **IV. Sample Collection**

#### **A. Field Blank and Sample Collection using a direct bottle immersion into the effluent flow:**

1. DH opens the cooler and removes plastic bags containing gloves and masks.
2. CH and DH put on dust masks first and then gloves.
3. DH opens cooler and removes the bag containing the field blank and sample bottles. DH opens the outer bag.
4. CH opens the inner bag and removes the field blank bottle.
5. CH fills the field blank bottle with distilled water supplied by the laboratory. This constitutes the collection of the field blank.
6. The CH immediately caps the sample bottle tightly and places it in the inside plastic bag. CH seals the inner bag.
7. DH holds the outside bag open and CH places the inside bag with the sample, into it.
8. DH reseals the outside bag. The sample is then placed into the storage cooler.
9. The process is repeated for collection of the effluent sample
10. Sample collection information is recorded on chain of custody records.
11. Sampling site is cleaned and the sample is transported to the lab.

## **B. Field Blank and Sample Collection using a sampling pole with bottle support.**

1. DH opens the cooler and removes plastic bags containing gloves and masks.
2. CH and DH put on dust masks first and then gloves.
3. DH opens the outer wrapping around the sample pole and bottle support.
4. CH opens the inner wrapping and assembles the pole and support (if two pieces) and leaves the pole covered with the inner plastic wrap until after the field blank collection and until the time of sample collection. **Do not delay this process.**
5. DH opens the cooler and removes the outer bag containing the field blank bottle.
6. CH opens the inner bag and removes the bottle. With CH holding the cap in one hand and the field blank bottle in the other, DH holds the distilled water container and fills the bottle held by CH with distilled water. This constitutes the field blank.
7. CH immediately tightly caps the field blank sample bottle and places it in the inside plastic bag. CH seals the inner bag.
8. DH holds the outer bag open and CH places the inner bag with the field blank sample into the outer bag.
9. DH reseals the outer bag. The field blank sample is then placed into the storage cooler.
10. DH opens the cooler and removes the outer bag containing the sample bottle.
11. CH opens the inner bag and removes the sample bottle and secures it into position in the bottle support on the pole while under the protective plastic.
12. CH inserts the sample bottle into the effluent and collects the sample and then places the sample bottle into the inner bag.
13. DH opens the outer bag.
14. CH places the inner bag with the sample into the outer bag.
15. Sample collection information is recorded on chain of custody records.
16. Sampling site is cleaned and the sample is transported to the lab.

## **C. Field blank using a peristaltic pump:**

1. DH opens the cooler and removes plastic bags containing gloves and masks.
2. CH and DH put on dust masks first and then gloves.
3. DH removes the lid from the laboratory provided distilled water for the field blank.
4. DH removes the bag containing the sample tubing from the cooler.
5. DH opens the bag.
6. CH removes the tubing and does not allow the tubing to touch anything.
7. CH removes the protective covering from the inlet side of the tubing.
8. CH lowers the weighted end of the tubing into the container of laboratory provided distilled water.
9. Once the inlet end of the tubing enters the distilled water container, DH holds the tubing.

10. DH inserts the flexible portion of the tubing into the peristaltic pump, while CH holds the discharge end of the tubing, protecting it from contamination.
11. CH removes the protective covering from the discharge end of the tubing and holds it over the purge container.
12. CH tells DH to turn the pump on.
13. CH holds the sample line over a purge container while purging for a minimum of five minutes.
14. After purging, CH tells DH to turn the pump off.
15. DH opens the cooler containing the double-bagged sample bottle.
16. DH opens the outside bag and does not touch the inside bag.
17. CH removes the inside bag (containing the sample bottle).
18. CH opens the bag and sample bottle.
19. CH holds the discharge end of the tubing over the sample bottle and tells DH to turn the pump on.
20. Once the sample bottle is filled, CH tells DH to turn the pump off.
21. CH tightly caps the sample bottle and reseals it in the inside plastic bag.
22. DH holds the outside bag open and CH places the inside bag, with the sample, into it.
23. DH reseals the outside bag. The sample is then placed into the storage cooler.
24. Sample collection information is recorded on chain of custody records.

**D. Sample Collection using a peristaltic pump after the field blank has been collected:**

1. CH removes the inlet end of the tubing from the carboy of DI water and places it into the sample location.
2. DH holds the tubing and makes sure that the inlet end and sides of the tubing do not contact anything.
3. CH tells DH to turn the pump on.
4. CH thoroughly rinses the tubing with the sample being collected.
5. CH tells DH to turn off the pump.
6. DH opens the cooler containing the double-bagged sample bottle.
7. DH opens the outside bag and does not touch the inside bag.
8. CH removes the inside bag (containing the sample bottle).
9. CH opens the bag and sample bottle.
10. CH holds the discharge end of the tubing over the sample bottle and tells DH to turn the pump on.
11. Once the sample bottle is filled, CH tells DH to turn the pump off.
12. CH tightly caps the sample bottle and reseals it in the inside plastic bag.
13. DH holds the outside bag open and CH places the inside bag, with the sample, into it.
14. DH reseals the outside bag. The sample is then placed into the storage cooler.
15. Sample collection information is recorded on chain of custody records.
16. Sampling site is cleaned and the sample is transported to the lab.

**Note: If a discrepancy between this procedure and the requirements of EPA Methods 1631E and 1669 exists, the method will prevail. Methods 1631E and 1669 are performance-based. Alternative procedures may be used, so long as those**

**procedures are demonstrated to yield reliable results. Alternative procedures must be demonstrated to produce acceptable mercury data within the specifications of the EPA Method 1631E prior to first use in the field. Documentation supporting the absence of contamination must be retained on file at the testing laboratory, certified cleaning contractor or field sampling facility and must be available for review by the Department upon request.**